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IS 13067 (1991): Impregnants for power capacitors [ETD 3: Fluids for Electrotechnical Applications]



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भारतीय मानक

पावर संधारित्रों के संसेचन — विशिष्ट

Indian Standard

**IMPREGNANTS FOR POWER CAPACITORS —
SPECIFICATION**

UDC 621.315.615 : 621.319.4

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Fluids for Electrotechnical Applications Sectional Committee had been approved by the Electrotechnical Division Council.

This standard is intended to cover requirements of insulating liquids as received by users and used as impregnants for capacitors.

The requirements of capacitors are covered by IS 2834 : 1986 'Shunt capacitors for power systems (*second revision*)'. In the manufacture of such capacitors insulating liquids are used as impregnants.

The requirements of electrical grade castor oil used in medium voltage power capacitors and high voltage energy storage capacitors are given in IS 9960 : 1981 'Electrical grade castor oil for use, as capacitor impregnant'.

In preparing this standard, considerable assistance has been derived from ASTM D 92 - 1985 'Standard test method for flash and fire points by Cleveland open cup' and ASTM D 2441 - 1985 'Standard test method for hydrolyzable chlorine compounds in chlorinated aromatic hydrocarbons (Askarels) by refluxing', issued by the American Society for Testing and Materials (ASTM).

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this standard.

Indian Standard

IMPREGNANTS FOR POWER CAPACITORS — SPECIFICATION

1 SCOPE

1.1 This standard covers the requirements and methods of tests for liquids used as impregnants in power capacitors conforming to IS 2834 : 1986.

2 REFERENCES

2.1 The referred Indian Standards are given in Annex A.

3 TERMINOLOGY

3.0 For the purpose of this standard, the following definitions shall apply.

3.1 Specific Gravity

The ratio of the weight in air of a given volume of the fluid at 29.5°C to the weight in air of an equal volume of water at 29.5°C.

3.2 Viscosity

3.2.1 Dynamic Viscosity

Dynamic viscosity of a fluid is the tangential force on a unit area either of two parallel planes at unit distance apart when the space between the plane is filled with fluid and one of the planes moves relatively to the other with unit velocity in its own plane.

3.2.2 Kinematic Viscosity

Kinematic viscosity of a fluid is equal to the quotient of dynamic viscosity and density.

3.3 Coefficient of Thermal Expansion

The coefficient of thermal expansion of liquid is the increase in volume per degree change in temperature. It is commonly stated as the average coefficient over a given temperature range.

3.4 Pour Point

The lowest temperature expressed as a multiple of 3°C at which the oil is observed to flow when cooled and examined under prescribed conditions.

3.5 Flash Point

The temperature at which the oil gives off so much vapour that this vapour when mixed with air forms an ignitable mixture and gives a momentary flash on application of a small pilot flame under the prescribed conditions of test.

3.6 Neutralization Value

The number of milligrams of potassium hydroxide required to neutralize all acidic constituents present in one gram of oil under prescribed conditions.

3.7 Specific Heat

The quantity of heat required to raise the temperature of one gram of the substance by one degree centigrade.

3.8 Thermal Conductivity

Time rate of heat flow through unit area of a homogenous substance under the influence of unit temperature gradient in the direction perpendicular to the area.

3.9 Permittivity

The permittivity of liquid is the ratio of capacitance C_p of a given configuration of electrodes with the material as dielectric to capacitance C_v of the same electrode with air as dielectric.

3.10 Resistivity

The resistivity (specific resistance) in ohm centimetres of a liquid is the ratio of potential gradient in volts per centimetre paralleling the current flow within the specimen to the current density in amperes per square centimetre at a given instant of time under prescribed conditions. This is numerically equal to the resistance between opposite faces of a 1 cm cube of a liquid.

3.11 Dielectric Dissipation Factor (Tangent Delta)

It is the tangent of the angle (δ) by which the phase difference between applied voltage and resulting current deviates from $\pi/2$ radian, when the dielectric of the capacitor consists exclusively of the insulating oil.

3.12 Repeatability

A quantitative measure of the variability associated with a single operator in a given laboratory, obtaining successive repeated result on the same apparatus. It is defined as that difference between two such single results that would only be exceeded in the long run in one case, in twenty in the normal and correct operation of the test method.

3.13 Reproducibility

A quantitative measure of variability associated with operators working in different laboratories,

each obtaining single result on identical test material. It is defined as that difference between two such single and independent test results that would be exceeded in the long run only in one case, in twenty in the normal and correct operation of test method.

4 REQUIREMENTS

4.1 General

The capacitor impregnants shall be based on ester compounds synthesized hydrocarbon compounds or their blends. It shall be free from sediment and suspended matter. The fluid should be compatible with the other materials for example polypropylene paper and aluminium foils used in capacitors.

4.2 The characteristics of the oil when it is sampled and tested in accordance with the methods referred to in Table 1 shall comply with the requirements laid down.

4.3 The impregnants should be non-toxic and should be bio-degradable.

4.4 Vapour Pressure

The vapour pressure of the impregnants should be suitable to facilitate impregnation of capacitors under vacuum.

5 SAMPLING

5.1 Sampling of the impregnants shall be done in accordance with IS 6855 : 1973.

6 REAGENTS

6.1 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1977) shall be used.

NOTE — 'Pure chemicals' shall mean, chemicals that do not contain impurities which affect the test results.

7 PACKING AND MARKING

7.1 The impregnants shall be stored in steel drums of 5, 20 and 210 litres capacity according to the requirement. The drums shall be effectively sealed to exclude moisture.

7.2 Marking

7.2.1 The drums shall be indelibly marked with the following:

- Indication of the source of manufacture,
- Quantity in litres,
- Name of the material,
- Country of manufacture, and
- Identification in code or otherwise to enable the date and lot of manufacture to be traced back to the factory records.

7.2.2 The containers may also be marked with the Standard Mark.

Table 1 Characteristics
(Clause 4.2)

Sl No.	Property	Esters	Synthesized Hydrocarbon	Cl Reference as per IS
1.	Colour in union (Max)	0.5	0.5	IS 548 (Part 1) : 1964
2.	Specific gravity at 29.5°C	0.9 — 1.2	0.9 — 1.1	IS 548 (Part 1) : 1964
3.	Refractive index at ambient temperature	1.48 — 1.51	1.55 — 1.57	IS 548 (Part 1) : 1964
4.	Coefficient of thermal expansion, $\text{Max./}^\circ\text{C}$	8.5 — 10 ⁻⁴	8 × 10 ⁻⁴	IS 9960 : 1981 (Annex A)
5.	Neutralization value in mg KOH/g, Max	0.01	0.02	IS 1448 (Part 2) : 1967
6.	Ash content, percent, Max	0.00	0.00	IS 9960 : 1981 (Annex C)
7.	Water content, ppm, Max	50	50	IS 2362 : 1973
8.	Kinematic viscosity, centistokes, Max			IS 1448 (Part 25) : 1976
	At 20°C	85	15	
	At 90°C	10	3.5	
9.	Pour point, °C, Max	—9	—24	IS 1448 (Part 10) : 1970
10.	Flash point, °C, Min	150	140	IS 1448 (Part 21) : 1970
11.	Specific heat at 20°C			
12.	Thermal conductivity	Under consideration		
13.	Gas absorption	Under consideration		
14.	Dielectric constant, Max			
	At 20°C	6.5	3.5	IS 6262 : 1971
	At 90°C	5.2	3.2	
15.	Chlorine content, ppm, Max	10	10	Annex B
16.	Biodegradability	—	—	Under consideration
17.	Fire point, °C, Min	220	150	Annex C
18.	Dielectric dissipation factor at 90°C, Max, (Tan Delta) at 50 Hz	0.03	0.002	IS 6262 : 1971
19.	Volume resistivity at 90°C, ohm-cm, Min	0.5 × 10 ¹³	10 × 10 ¹³	IS 6103 : 1971
20.	Dielectric strength, kV (2.5 mm gap), Min	40	60	IS 6792 : 1972
21.	Ageing 96 h, 115°C without metal catalyst			IS 12177 : 1987
	1. Dissipation factor at 90°C, Max	0.05	0.003	IS 6262 : 1971
	2. Volume resistivity, ohm-cm, at 90°C, Min	1 × 10 ¹¹	1.5 × 10 ¹³	IS 6103 : 1971
22.	Corrosive sulphur	←—Non-corrosive—→		IS 335 : 1983
23.	Vapour pressure	Under consideration		

ANNEX A

(Clause 2.1)

LIST OF REFERRED INDIAN STANDARDS

<i>IS No.</i>	<i>Title</i>	<i>IS No.</i>	<i>Title</i>
335 : 1983	Specification for new insulating oils (<i>third revision</i>)		(Part 25) Determination of kinematic and dynamic viscosity (<i>first revision</i>)
548 (Part 1) : 1964	Methods of sampling and test for oils and fats: Part 1 Sampling physical and chemical tests	2362 : 1973	Procedure basic climatic and durability tests for optical instruments
1070 : 1977	Water for general laboratory use	2834 : 1986	Shunt capacitors for power systems (<i>second revision</i>)
1448 (Part 2) : 1967	Methods of test for petroleum and its products leading pages to IS 1448 (Part 2) Acidity (<i>first revision</i>)	6103 : 1971	Method of test for specific resistance (resistivity) of electrical insulating liquids
1448 (Part 10) : 1970	Methods of test for petroleum and its products leading pages to IS 1448 (Part 10) Cloud point and pour point (<i>first revision</i>)	6262 : 1971	Method of test for power factor and dielectric constant of electrical insulating liquids
1448 (Part 21) : 1970	Methods of test for petroleum and its products leading pages to IS 1448 (Part 21) Flash point (closed) by Pensky Martens apparatus (<i>first revision</i>)	6792 : 1972	Method for determination of electric strength of insulating oils
1448 (Part 25) : 1976	Methods of test for petroleum and its products leading pages to IS 1448	9960 : 1961	Electrical grade castor oil for use as capacitor impregnant
		12177 : 1987	Methods of test for oxidative ageing of electrical insulation of petroleum oils by the open beaker method

ANNEX B

(Table 1)

TEST METHOD FOR CHLORINE CONTENT IN CAPACITORS IMPREGNANTS

B-1 SCOPE

B-1.1 This test method covers the determination of chlorine compounds in capacitors impregnants.

B-1.2 This standard may involve hazardous materials, operation, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever used this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

B-2 SUMMARY OF METHOD

B-2.1 This test method uses a quantitative potentiometric titration using silver nitrate in an essentially nonaqueous medium to determine chloride ions. These are obtained after metha-

nolic sodium hydroxide hydrolysis of chlorine compounds in capacitor impregnating fluids.

B-3 SIGNIFICANCE AND USE

B-3.1 The measured chloride ion, reported as parts per million in the sample, is indicative of free and hydrolysable chlorine compounds that would lead to corrosion under prescribed conditions of test.

B-3.2 Refluxing with methanolic sodium hydroxide offers the detection of certain chlorine addition compounds that could present as impurities and do not respond fully to hydrolysts at the lower temperature.

B-4 INTERFERENCE

B-4.1 The presence of compounds containing hydrolysable halogen other than chlorine, that

is, bromine and iodine, will give positive results, as will also any other foreign substance capable of forming an insoluble compound with silver nitrate.

B-4.2 The amount of chloride ions measured when the test is applied to pure impregnants is very small, ranging near 1 ppm. Therefore, the test must be made in area where the atmosphere is free from contaminating influence and it is essential to make a blank determination. The value obtained on the blank will indicate the degree to which the test environment, reagents, and glassware are satisfactory for this type of testing.

B-5 APPARATUS

B-5.1 Flask, borosilicate glass, 500-ml, with 24/40 standard-taper joint.

B-5.2 Reflux condensor, with mated 24/40 standard-taper joint.

B-5.3 Combination magnetic stirrer and hot plate.

B-5.4 Magnetic stirring bar.

B-5.5 Microburette, 1.0-ml capacity, graduated in 0.01-ml divisions.

B-5.6 Electrodes, silver and glass.

B-5.7 Potentiometer, suitable for use with the glass electrode.

B-6 REAGENTS

B-6.1 Purity of Reagents

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the relevant specifications where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

B-6.2 Purity of Water

Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to IS 1070 : 1977. In addition, the water shall be chloride-free, as detected by this test.

B-6.3 Acetone, Chloride-free

Prepare chloride-free acetone by distillation from silver nitrate, as described in B-6.4 for methanol. Determine its freedom from chlorides by potentiometric titration. A chloride content equivalent to less than 0.01 ml of 0.0025 N silver nitrate solution per 100 ml should be derived.

B-6.4 Chloride-free Methanol

Reflux 5 litres of methanol with 0.5 g of silver nitrate for 30 min. (Chloride-free methanol

may be obtained also by distillation over sodium hydroxide). Distill the methanol from the silver nitrate, discarding the first 100 ml to flush the apparatus. Continue the distillation till about 90 percent of the methanol has been distilled from the flask. Discard the remaining 10 percent. Check the methanol by titration to assure its purity. The chloride ion concentration should be equivalent to less than 0.02 ml of 0.0025 N silver nitrate solution per 100 ml of methanol.

B-6.5 Standard Silver Nitrate Solution (0.0025 N)

Weigh accurately 0.4247 g of silver nitrate. Transfer it to a 1-l volumetric flask. Add water to dissolve silver nitrate. Add 3.0 ml of concentrated nitric acid (HNO_3 , sp. gr. 1.42) and then add water to the 1 l mark. Standardize this solution against a pure chloride standard. Check the solution at least monthly to assure a consistent strength of reagent.

B-6.6 Sodium Hydroxide-Methanol Solution (4 g/l)

Dissolve 4.0 g of sodium hydroxide in 1 litre of chloride-free methanol (B-6.4).

B-6.7 Sulphuric Acid 1:1 (v/v)

With constant stirring pour 1 volume of concentrated sulphuric acid (H_2SO_4 sp. gr. 1.84) into 1 volume of water.

B-7 PROCEDURE

NOTE — Because of the very small amount of chloride determined in pure impregnants, be sure, that during the test, contamination does not occur from other sources of halogen such as, from the laboratory atmosphere or through contact with the human skin. The test should not be made in a laboratory where strong halogen solutions are handled, stored, or evaporated. All glassware, apparatus, and the area in which the test is run must be analytically clean.

B-7.1 Weigh 100 g into a tared 500-ml flask to the nearest 0.1 g on a suitable balance.

B-7.2 Using clean dry tongs, place the magnetic stirring bar in the flask.

B-7.3 Add by graduate cylinder, 100 ml of 0.1 N methanolic NaOH solution.

B-7.4 Attach the reflux condensor and reflux vigorously for 1 h. Measure the reflux time from the start of boiling.

B-7.5 Turn off the heat and place a container of cold water under the flask to cool the sample.

B-7.6 Rinse down the condenser with acetone and quantitatively transfer the solution into a 400-ml beaker, using a total of 100 ml of acetone.

B-7.7 Add 3 ml of sulphuric acid 1:1 (v/v) to the sample from a pipette.

B-7.8 Place the sample beaker on the magnetic stirrer. Stir the sample, insert the silver-glass electrode system, and titrate with 0.0025 N silver nitrate solution, using the microburet.

B-7.8.1 Normal samples of capacitor impregnants require extremely small amounts of silver nitrate solution. Therefore, when titrating use 0.05-ml additions of the silver nitrate solution and allow sufficient time for equilibrium to be established before recording the emf change. If three or four additions of 0.05-ml increments of silver nitrate solution give a potential change of less than 1 mV/0.05 ml of solution, add larger amounts such as 0.01 ml until such a change is observed. Then reduce the additions again to 0.05 ml to complete the titration. The end point is the point where the greatest potential change per 0.05 ml increment of silver nitrate solution is obtained.

B-7.9 Make a blank determination exactly as above, omitting the sample.

B-8 CALCULATION

B-8.1 Calculate the amount of chlorine as follows:

Chlorine, ppm = $[(A - B) N / C] \times 35.46 \times 10^3$
where

A = millilitres of silver nitrate solution required for titration of the sample;

B = millilitres of silver nitrate solution required for titration of the blank;

N = normality of the silver nitrate solution; and

C = grams of sample used.

B-9 REPORT

B-9.1 Report chlorine content (average of two duplicate determinations) to the nearest integral part per million. If the average of the two determinations is less than 1 ppm, report the chlorine content as 1 ppm.

B-10 PRECISION

B-10.1 Checking Limit for Duplicates

With 95 percent probability, duplicate determination on the same sample should be considered suspect if they differ by more than 0.1 ppm.

B-10.2 Reproducibility

Two laboratories each reporting the average of duplicate determinations to the nearest integral part per million should agree exactly.

B-10.3 The precision noted in B-10.1 and B-10.2 is based on the results of a round-robin test conducted by six laboratories.

ANNEX C (Table 1)

TEST METHOD FOR FLASH AND FIRE POINTS BY CLEVELAND OPEN CUP

C-1 SCOPE

C-1.1 The test method covers determination of the flash and fire points of all petroleum products except fuel oils and those having an open cup flash below 79°C.

C-1.2 This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

C-1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and

health practices and determine the applicability of regulatory limitations prior to use.

C-2 DEFINITIONS

C-2.1 Flash Point

The lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg) at which application of a test flame causes the vapour of a specimen to ignite under specified conditions of test.

NOTES

1 The material is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the specimen.

2 Occasionally, particularly near the actual flash point, the application of the test flame will cause a blue halo or enlarged flame this is not a flash and should be ignored.

C-2.2 Fire Point

The lowest temperature at which a specimen will sustain burning for 5 sec.

C-3 SUMMARY OF METHOD

C-3.1 The test cup is filled to a specified level with the sample. The temperature of the sample is increased rapidly at first and then at a slow constant rate as the flash point is approached. At specified intervals a small test flame is passed across the cup. The lowest temperature at which application of the test flame causes the vapours above the surface of the liquid to ignite is taken as the flash point. To determine the fire point, the test is continued until the application of the test flame causes the oil to ignite and burn for at least 5 sec.

C-4 SIGNIFICANCE AND USE

C-4.1 Flash point measures the tendency of the sample to form a flammable mixture with air under controlled laboratory conditions. It is only one of a number of properties that must be considered in assessing the overall flammability hazard of a material.

C-4.2 Flash point is used in shipping and safety regulations to define 'flammable' and 'combustible' materials. One should consult the particular regulation involved for precise definitions of these classes.

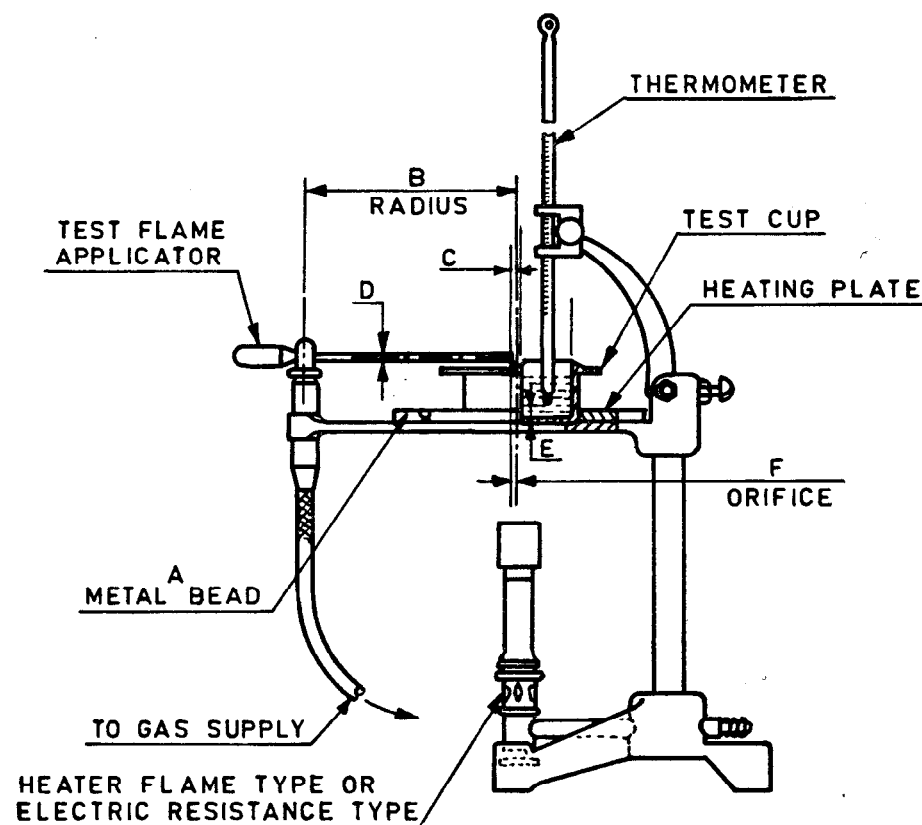
C-4.3 Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively non-volatile or non-flammable material.

C-4.4 Fire point measures the characteristics of the sample to support combustion.

C-5 APPARATUS

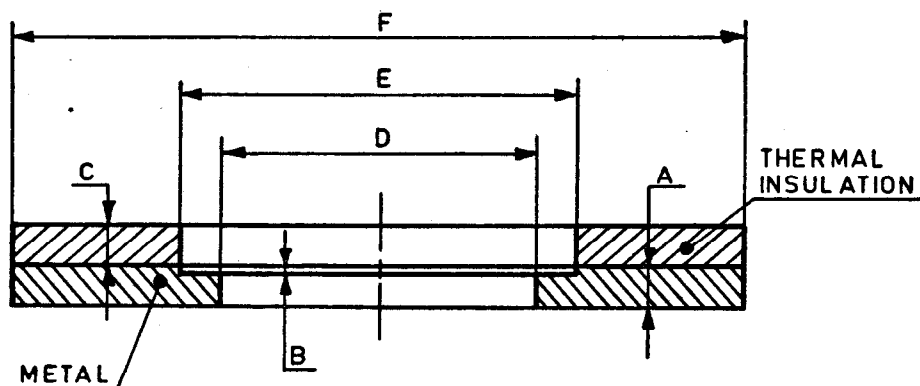
C-5.1 Cleveland Open Cup Apparatus

This apparatus consists of the test cup, heating plate, test flame applicator, heater, and supports described in detail as follows. The assembled apparatus heating plate and cup are illustrated in Fig. 1, 2 and 3 respectively.



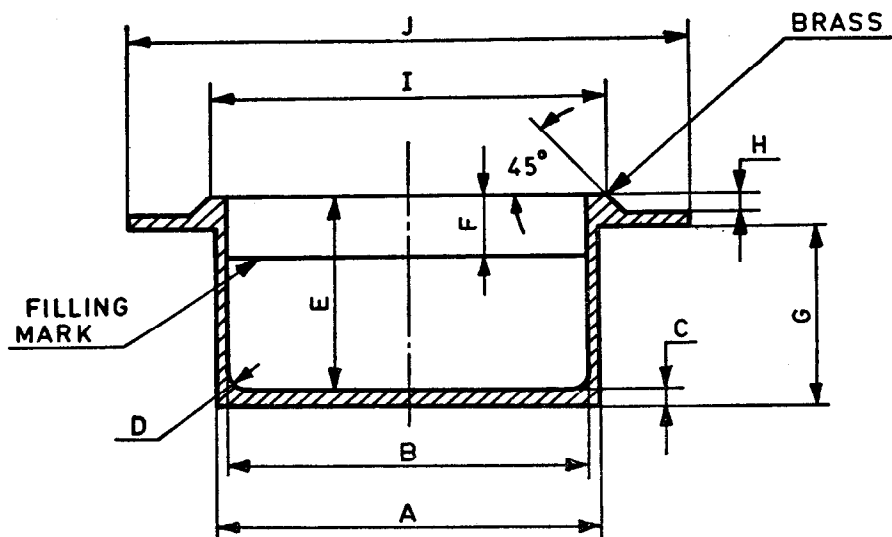
	Millimetres	
	Min	Max
A — Diameter	3.2	4.8
B — Radius	152	Nominal
C — Diameter	1.6	Nominal
D		2
E	6	7
F — Diameter	0.8	Nominal

FIG. 1 CLEVELAND OPEN CUP APPARATUS



	Millimetres	
	<i>Min</i>	<i>Max</i>
<i>A</i>	6	7
<i>B</i>	0·5	1·0
<i>C</i>	6	7
<i>D</i> — Diameter	55	56
<i>E</i> — Diameter	69·5	70·5
<i>F</i> — Diameter	146	159

FIG. 2 HEATING PLATE



	Millimetres	
	<i>Min</i>	<i>Max</i>
<i>A</i>	67·5	69
<i>B</i>	63	64
<i>C</i>	2·8	3·5
<i>D</i> — Radius	4	Nominal
<i>E</i>	32·5	34
<i>F</i>	9	10
<i>G</i>	31	32·5
<i>H</i>	2·8	3·5
<i>I</i>	67	70
<i>J</i>	97	100

FIG. 3 CLEVELAND OPEN CUP

C-5.1.1 Test cup conforming to Fig. 3 with dimensions as shown shall be used. The cup shall be made of brass or other non-rusting metal of equivalent heat conductivity. The cup may be equipped with a handle.

C-5.1.2 Heating Plate

A brass cast iron, wrought iron or steel plate with a centre hole surrounded by an area of plane depression, and a sheet of hard asbestos board which covers the metal plate except over the area of plane depression in which the test cup is supported. The essential dimensions of the heating plate are shown in Fig. 2, however, it may be square instead of round, and the metal plate may have suitable extensions for mounting the test flame applicator device and the thermometer support. Also a metal bead, as mentioned in C-5.1.3 may be mounted on the plate so that it extends through and slightly above a suitable small hole in the asbestos board.

C-5.1.3 Test Flame Applicator

The device for applying the flame may be of any suitable type, but it is suggested that the tip be approximately 1.6 mm in diameter at the end, and that the orifice be 0.8 mm in diameter. The device for operating the test flame may be mounted in such a manner as to permit automatic duplication of the sweep of the test flame, the radius of swing being not less than 150 mm and the centre of the orifice being supported so that it swings in a plane not greater than 2 mm above the plane of the rim of the cup. It is desired that a bead having a diameter of 3.2 to 4.8 mm be mounted in a convenient position on the apparatus so that the size of the test flame can be compared to it.

C-5.1.4 Heater

Heat may be supplied from any convenient source. The use of a gas burner of alcohol lamp is permitted, but under no circumstances are products of combustion or free flame to be allowed to come up around the cup. An electric heater controlled by a variable voltage transformer is preferred. The source of heat shall be centered under the opening of the heating plate with no local superheating. Flame type heaters may be protected from drafts or excessive radiation by any suitable type of shield that does not project above the level of the upper surface of asbestos board.

C-5.1.5 Thermometer Support

Any convenient device may be used which will hold the thermometer in the specified position during a test and which will permit easy removal of thermometer from the test cup upon completion of a test.

C-5.1.6 Heating Plate Support

Any convenient support which will hold the heating plate level steady may be employed.

C-5.2 Shield

A shield of 460 mm square and 610 mm high and having an open front is recommended.

C-5.3 Thermometer

A thermometer having a range of 90 to 360°C shall be used.

NOTE — There are automatic flash point testers available and in use which may be advantageous in the saving of testing time, permit the use of smaller samples and have other factors which may merit their use. If automatic testers are used, the user must be sure that all of the manufacturer's instructions for calibrating, adjusting, and operating the instrument are followed. In case of dispute the flash point as determined manually shall be considered the reference test.

C-6 SAFETY PRECAUTIONS

C-6.1 The operator must exercise and take appropriate safety precautions during the initial applications of the test flame, since samples containing low-flash material may give an abnormally strong flash when the test flame is first applied.

C-7 SAMPLING

C-7.1 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily and make a transfer unless the sample temperature is at least the equivalent of 10°C below the expected flash point. Do not use samples from leaky containers for this test.

C-7.2 Do not store samples in plastic (polyethylene, polypropylene, etc) containers since volatile material may diffuse through the walls of the enclosure.

C-7.3 Light hydrocarbons may be present in the form of gases, such as propane or butane and may not be detected by testing because of losses during sampling and loading of the test apparatus. This is especially evident on heavy residues or asphalt from solvent extraction processes.

C-8 PREPARATION OF APPARATUS

C-8.1 Support the apparatus on a steady level table in a draft-free room or compartment. Shield the top of the apparatus from strong light by any suitable means to permit ready detection of the flash point. Tests made in a laboratory hood (see Note below) or in any location where drafts occur are not to be relied upon. During the last 17°C rise in temperature prior to the flash point, care must be taken to avoid disturbing the vapours in the test cup by careless movement or breathing near the cup.

NOTE — With some samples whose vapours or products of pyrolysis are objectionable, it is permissible to place the apparatus with shield in a

hood, the draft of which is adjustable so that vapours may be withdrawn without causing air currents over the test cup during the final 56°C rise in temperature prior to the flash point.

C-8.2 Wash the test cup with an appropriate solvent to remove any oil or traces of gum or residue remaining from a previous test. If any deposits of carbon are present, they should be removed with steel wool. Flush with cold water and dry for a few minutes over an open flame or a hot plate to remove the last traces of solvent and water. Cool the cup to at least 56°C below the expected flash point before using.

C-8.3 Support the thermometer in a vertical position with the bottom of the bulb 6.4 mm from the bottom of the cup and locate at a point half way between the centre and side of the cup on a diameter perpendicular to the arc (or line) of the sweep of the test flame and on the side opposite to the test flame burner arm.

NOTE — The immersion line engraved on the thermometer will be 2 mm below the level of the rim of the cup when the thermometer is properly positioned.

C-9 PROCEDURE

C-9.1 Fill the cup at any convenient temperature (see Note below) so that the top of the meniscus is exactly at the filling line. If too much sample has been added to the cup, remove the excess, using a dropper, however, if there is sample on the outside of the apparatus, empty, clean, and refill it. Destroy any air bubbles on the surface of the sample.

NOTE — Viscous samples should be heated until they are reasonably fluid before being poured into the cup, however, the temperature during heating must not exceed 56°C below the probable flash point.

C-9.2 Light the test flame and adjust it to a diameter of 3.2 to 4.8 mm, the size of the comparison bead if one is mounted on the apparatus.

C-9.3 Apply heat initially so that the rate of temperature rise of the samples is 14 to 17°C/min. When the sample temperature is approximately 56°C below the anticipated flash point, decrease the heat so that the rise of temperature rise of the last 28°C before the flash point is 5 to 6°C/min.

C-9.4 Starting at least 28°C below the flash point, apply the test flame when the temperature read on the thermometer reaches each successive 2°C mark. Pass the test flame across the centre of the cup, at right angles to the diameter which passes through the thermometer. With a smooth, continuous motion apply the flame either in a straight line or along the circumference of a circle having a radius of at least 150 mm. The centre of the test flame must move in a horizontal plane not more than 2 mm above the plane of the upper edge of the cup and passing in one direction only. At the time of the next

test flame application, pass the flame in the opposite direction. The time consumed in passing the test flame across the cup in each case shall be about 1 sec.

NOTE — Meticulous attention to all details relating to the test flame applicator, size of the test flame, rate of temperature increase, and rate of passing the test flame over the sample is necessary for good results.

C-9.5 Record as the observed flash point the temperature read on thermometer when a flash appears at any point on the surface of the oil, but do not confuse the true flash with the bluish halo that sometimes surrounds the test flame.

C-9.6 To determine the fire point, continue heating so that the sample temperature increases at a rate of 5 to 6°C/min. Continue the application of the test flame at 2°C intervals until the oil ignites and continues to burn for at least 5 sec. Record the temperature at this point as the observed fire point of the oil.

C-10 CALCULATION AND REPORT

C-10.1 Observe and record the barometric pressure at the time of the test. When the pressure differs from 760 mm Hg, correct the flash or fire point, or both, by means of the following equations:

$$\begin{aligned} &\text{Corrected flash or fire point or both} \\ &= C + 0.03 (760 - P) \end{aligned}$$

where

C = observed flash point, or both, to the nearest 2°C, and

P = barometric pressure, mm Hg.

C-10.2 Record the corrected flash or fire point, or both, to the nearest 2°C.

C-10.3 Report the recorded flash or fire point value, or both, as the COC flash or fire point, or both.

C-11 PRECISION

C-11.1 The following data should be used for judging the acceptability of results (95 percent confidence).

C-11.1.1 Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

	Repeatability
Flash point	8°C
Fire point	8°C

C-11.1.2 The result submitted by each of two laboratories should be considered suspect if the results differ by more than the following amounts:

	Reproducibility
Flash point	17°C
Fire point	14°C

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